

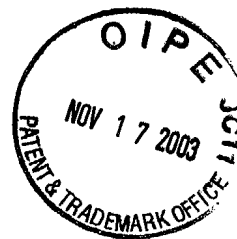
IN THE UNITED STATES PATENT & TRADEMARK OFFICE

In re Patent Application of W. Friedrich et al.

Serial No. 09/235,242

Filed: January 22, 1999

For: Process for preparing gamma-alkoxyamines



DECLARATION

I, Frank Haese, Dr. Ing., a citizen of Germany and residing at Hauptstraße 38 B ,D-24855 Bollingstedt, Germany, hereby declare as follows:

I am a fully trained chemist, having studied chemistry at the Technical University of Darmstadt, Germany, from 1989 to 1998.

I was awarded my doctor's degree by the Technical University of Darmstadt, Germany, in 1998.

Since 1998, when I joined BASF Aktiengesellschaft of D-67056 Ludwigshafen, Germany, I have been engaged in the development of catalysts and processes for the manufacture of organic products.

I have studied the subject matter disclosed and claimed in Application of Serial No. 09/235,242 filed on January 22, 1999 and am therefore familiar therewith.

I have read the Office Action dated May 12, 2003.

O'Lenick et al. (US 5,196,589), examples No. 1 and 10, have been regarded to be the closest prior art, also because these examples were performed in the absence of a free radical compound.

These examples 1 and 10 show an efficiency of reaction (by reduction of hydroxyl value) of 58.2 % and 68.2 %, respectively.

The solved technical problem underlying the invention was *not* to find *yet another* process, but to find an *improved* process for preparing a  $\gamma$ -alkoxyamine starting from an  $\alpha,\beta$ -unsaturated nitrile, without there being any need for any prior costly removal or destruction or neutralization of the catalyst used for the 1,4-addition of an alcohol to the  $\alpha,\beta$ -unsaturated nitrile, in which the product-amine is obtained in significantly *higher yield* as in the closest prior art.

Our inventive example 5 (Nos. 7 and 8 are also examples according to the invention) demonstrates the unexpected beneficial results (= higher yield of gamma-alkoxyamine) in the light of comparative example 5, which must be regarded as an example according to the above-mentioned closest prior art process.

It is my understanding that the Examiner asked for further side-by-side comparison examples demonstrating the superior results over examples No. 1 and 10 of O'Lenick et al.

The following supplemental experiments were conducted under my supervision. The significant difference between these supplemental experiments according to the invention and the examples No. 1 and 10 of O'Lenick et al. lies in the catalyst used for the addition of the alcohol to the acrylonitrile (DBU vs. potassium hydroxide). Since the controlling factor is the addition-reaction when looking at the reduction of hydroxyl value, the kind of catalyst used for the hydrogenation of the addition-product is not important in this context.

#### Experiment report:

##### Example 9:

150 g (0.0448 Mol) of polyethylene glycol (molecular weight: 3350) and 4.5 g of DBU (= 1,8-diazabicyclo[5.4.0]undec-7-ene) (0.029 Mol) were introduced as initial charge and gradually admixed at 25 °C with 4.8 g (0.090 Mol) of acrylonitrile added at 10 %/min. The mixture was subsequently reacted at 60 °C for further 72 hours. The cyanoethylated polyethylene glycol was then continuously hydrogenated in acetic acid at 20 °C over a PtO<sub>2</sub> catalyst with two equivalents of hydrogen with a hydrogen partial pressure of about 0.1 MPa.

The conversion of polyethylene glycol was 93 %, which corresponds to an efficiency of reaction (by reduction of hydroxyl value) of 93 %.

##### Example 10:

The example was performed as described above, with the difference that 7.5 g of DBU (0.049 Mol) were used.

The conversion of polyethylene glycol was 100 %, which corresponds to an efficiency of reaction (by reduction of hydroxyl value) of 100 %.

Example 11:

135.5 g (0.502 Mol) of stearyl alcohol and 4.05 g of DBU (= 1,8-diazabicyclo[5.4.0]undec-7-ene) (0.026 Mol) were introduced as initial charge and gradually admixed at 25 °C with 27 g (0.509 Mol) of acrylonitrile added at 10 %/min. The mixture was subsequently reacted at 60 °C for further 72 hours. The cyanoethylated stearyl alcohol was then continuously hydrogenated in acetic acid at 20 °C over a PtO<sub>2</sub> catalyst with two equivalents of hydrogen with a hydrogen partial pressure of about 0.1 MPa.

The conversion of stearyl alcohol was 78 %, which corresponds to an efficiency of reaction (by reduction of hydroxyl value) of 78 %.

Example 12:

The example was performed as described above, with the difference that 6.77 g of DBU (0.044 Mol) were used.

The conversion of stearyl alcohol was 83 %, which corresponds to an efficiency of reaction (by reduction of hydroxyl value) of 83 %.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further that these statements are made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at D-67056 Ludwigshafen, Germany, this 7<sup>th</sup> day of November 2003.



Signature of Declarant